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Production of hydrogen from rice straw using microwave-induced pyrolysis

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HIGHLIGHTS

- H_2 gas produced from the pyrolysis of rice straw is measured at different powers.
- H_2, CO_2 and CO are the main gas products in the pyrolysis of rice straw.
- The optimum concentration of the hydrogen production is 56.08% at a power of 1000 W.

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ABSTRACT

This study investigated hydrogen produced by feeding rice straw into a microwave plasma system. The conversion rate is evaluated according to the concentration of hydrogen and other gas products. When feed rice straws into the microwave plasma system at 800 W, 900 W, and 1000 W using an upstream method, the concentrations of hydrogen production were 48%, 53%, and 56%, respectively. When feed rice straws by using the downstream method, the concentrations of hydrogen production are 34%, 40%, and 45%, respectively. These results indicate that the upstream feeding method is more favorable than the downstream for hydrogen production, and an increase of power can enhance the production of hydrogen. Optimal hydrogen production is achieved when rice straws are fed into the system using the upstream method at a power of 1000 W; each gram of rice straw produced approximately 40.47 mg of hydrogen (conversion rate = 67.45%).

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1. Introduction

Rice straw is a preferred bioenergy source because it is residual material resulting from rice production. The annual average of rice production was 1.6 million tons from 2001 to 2005 in Taiwan [1], and the annual average rice production from 2006 to 2011 in Pakistan is 5.93 million tones [2]. The International Rice Research Institute (IRRI) reported that the total rough rice production in the world was more than 600 million tones approximately in 2004 [3]. The use of rice straw not only saves the cost of disposal but also produces valuable bioenergy, achieving the goal of resource recovery and reuse. Biomass waste can be efficiently transformed into bioenergy by applying thermochemical methods such as combustion, pyrolysis, and gasification [4,5]. Among them, the pyrolysis process not only reduces the volume of waste considerably, but also enables the recovery of value-added products. However, because of the nature of its endothermic reactions, the pyrolysis process requires a continuous supply of heat to chemically decompose biomass.

Conventional heating methods include external heating by conduction, convection, and radiation [6]. However, these methods possess certain drawbacks, including heat transfer resistance, heat loss to surroundings, use of portions of heat supplied to biomass materials, and damage to reactor walls because of continuous electrical heating. Accordingly, a new technique of using microwaves as an alternative heating source was developed. Microwaves are currently used in several applications such as food drying and heating [7,8], chemical synthesis [9,10], digestion [11,12], and extraction [13,14]. Microwave plasma, commonly used in microwave ovens, diamond synthesis, and IC manufacturing, has the advantages of easy operation, electrodeless reactors, high plasma density, and high electron mean energy. Compared with conventional pyrolysis conducted using an electric furnace, microwave pyrolysis produces more H_2 and CO [15,16], which is the so-called syngas, or synthesis gas. In addition, microwave pyrolysis generates fewer polycyclic aromatic hydrocarbons (PAHs) and, therefore, produces fewer hazardous compounds [17,18].

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This study investigated the microwave-assisted pyrolysis of rice straw by using upstream and downstream feeding methods. Pyrolytic reactions of rice straw were implemented under different microwave powers in this study, different microwave powers present various decomposed temperatures of rice straw. Hence, the temperature profiles under different microwave power levels are studied in this manuscript. The products, pyrolysis temperature, and temperature increase rate were analyzed to obtain optimal conditions and to maximize hydrogen production.

2. Experimental section

The experimental setup, comprising an electrodeless microwave-excited atmospheric plasma system (APS) and a product analysis system, is shown schematically in Fig. 1. Details of this experimental setup are described in previous studies [19–21]. Experiments in the present study were repeated at least three times to provide evidence of reproducibility.

2.1. Pyrolysis procedure

The pyrolysis experiments were conducted in an atmospheric-pressure microwave plasma reactor at an applied microwave power of 800, 900, and 1000 W, corresponding to the temperatures of 1063, 1093, and 1121 K in the plasma zone. Approximately 1 g of rice straw at room temperature was fed into the reacting zone upstream of the cavity resonator, and N_2 was used as bath gas at a flow rate of 12 $\mathrm{l\,min^{-1}}$ (an axial flow rate of 3 $\mathrm{l\,min^{-1}}$ and a swirl flow rate of 9 $\mathrm{l\,min^{-1}}$). The flow rate of N_2, supplied from compressed gas tanks, was maintained constant. The pyrolysis of biomass was conducted in nitrogen at a pressure of 1 atm. The reactor was made from a quartz tube, which was 35 cm in length and had inner and outer diameters of 2.9 and 3.3 cm, respectively.

2.2. Rice straw and gas analysis

Both the pyrolysis characteristics and product distribution of rice straw were analyzed in this study. Morphological changes of the rice straw samples before and after pyrolysis were observed using environmental scanning electron microscopy (ESEM). Elemental chemical analysis (C, H, N, S, and O) of rice straw and the residue after pyrolysis was performed using an Elementary Vario Micro Cube elemental analyzer (EA). Sampling was performed by continually withdrawing gases from within the plasma zone using a micro probe with a diameter of approximately several hundred microns. Gas chromatographs (GC) equipped with capillary columns (type: SUPELCO 13821), a thermal conductivity detector (TCD), and a residual gas analyzer (RGA), were used for stable species measurements. Residual gas analyzer (RGA) provides detailed gas analysis of vacuum systems at about half the price of competitive models, the type of RGA is Extorr RGA200. Each RGA system contains a quadrupole probe, electronics control unit (ECU), and a real-time Windows software package that is used for data acquisition and analysis, as well as probe control. The RGA was used to monitor the hydrogen concentration produced throughout the entire pyrolysis process. Regarding the H_2 analyzed using the GC/TCD, the carrier gas was nitrogen and the detector oven and vaporizer temperatures were 513 and 383 K, respectively. The GC oven temperature was set to 383 K for 10 min, ramped to 473 K at 288 $\mathrm{K\,min^{-1}}$, and maintained for 14 min. Regarding the CO and CO_2 analyzed using the GC/TCD, the carrier gas was helium and the detector oven and vaporizer temperatures were 393 K and 323 K, respectively. The GC oven temperature was set to

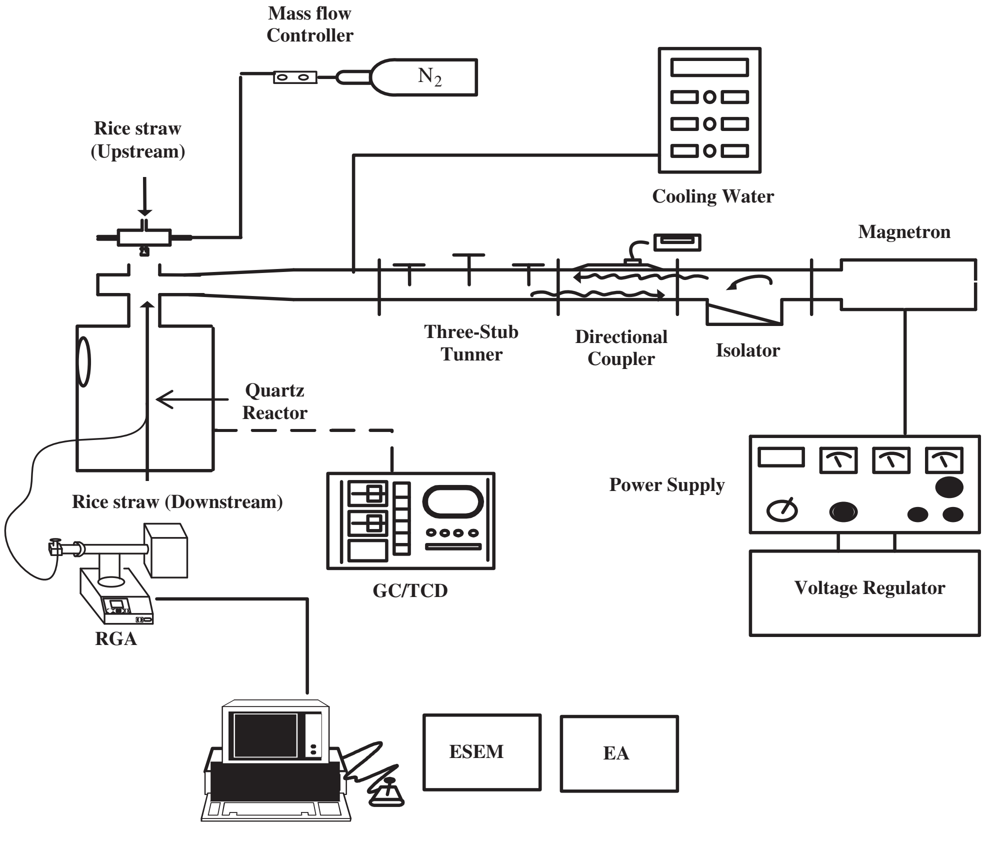


Fig. 1. Schematic of the atmospheric-pressure microwave plasma reactor.

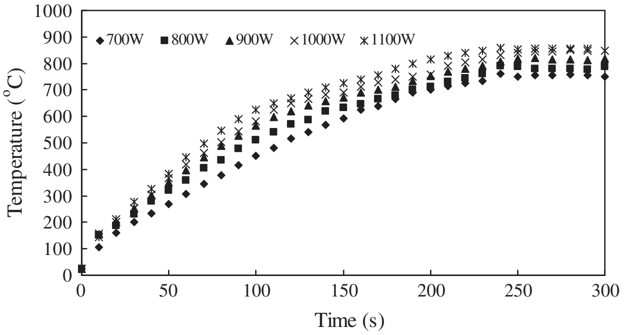


Fig. 2. The reaction temperature at various microwave power.

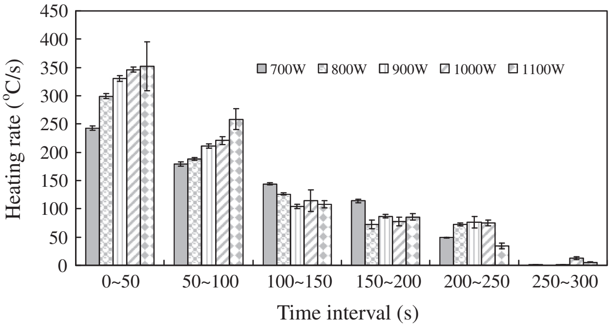


Fig. 3. Heating rates at various microwave plasma power.

333 K for 5 min, ramped to 498 K at 293 K min^{-1}, and maintained for 10 min.

3. Results and discussion

3.1. Temperature profiles

Fig. 2 shows the temperature profiles of pyrolytic reactions occurring under different microwave power levels. Between 0 and 300 s of microwave radiation, the reaction temperatures slowly increased, and the maximal reaction temperatures were only 700–900 °C under the microwave power of 700–1100 W. Regarding the microwave power of 900 W and higher, the reaction temperatures increased much faster. Finally, when a microwave power higher than 1100 W was applied, the maximal reaction temperatures were 240 s or less. These phenomena show that higher microwave power yields a higher heating rate. The maximal reaction temperature is summarized in Table 1. The heating rates at different time intervals and under various microwave powers are shown in Fig. 3. The data indicates that the increases in temperature were mainly concentrated in the first 50 s, especially for the microwave powers of 900–1100 W. Regarding 700–1100 W of microwave power applied in the first 50 s, the highest heating rates were 240–350 °C/min, and high microwave power (1100 W) produced a high heating rate. In the time interval of 50–100 s, the heating rates decreased to 180–260 °C/min. After 250 s, there was no substantial heating activity under the microwave powers of 700–1100 W. The high heating rates in the first 100 s may be due to the quick reactivity of microwave pyrolysis, and this heating behavior is similar to that described in the works of Monsef–Mirzai et al. [22], El Harfi et al. [23], and Menendez et al. [24]. The relationship between the microwave power (W) and the heating rate (dT/dt), as well as that between the microwave power and the maximal temperature (T_{max}), were also determined. Microwave power vs. heating rate shows a linear relationship and lists as the following equations,

$$dT/dt = 0.193\,\mathrm{W} + 37.9 \tag{1}$$

the slope is 0.193 and the intercept is 37.9, which indicates that to start an apparent rise in temperature, sufficient power input is needed. If $dT/dt = 0$, $W = 196$ W, which represents the minimal microwave power to raise the temperature. As shown in Fig. 4, the linear regression of microwave power and maximal temperature lists as the following equations,

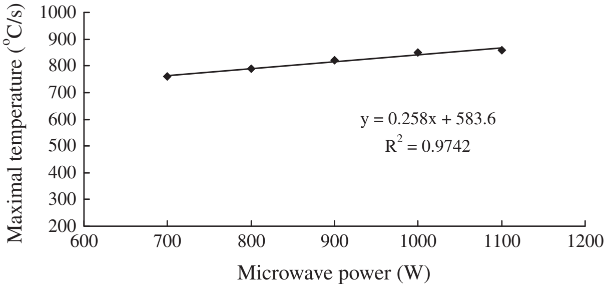


Fig. 4. The relationship of microwave plasma power vs. T_{max}.

$$T_{max} = 0.258\,\mathrm{W} + 583.6 \tag{2}$$

the slope is 0.258, the intercept is 583.6, and R^2 is 0.9742, implying that microwave power exhibits a strong relationship with the maximal temperature. These relationships validate the reliability of the temperature measurements and experimental results.

3.2. Elemental analysis of rice straw

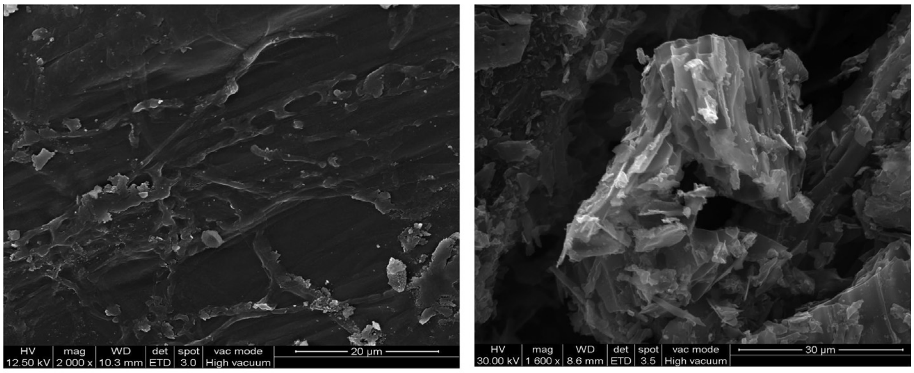
Table 2 shows the elemental analysis of rice straw before and after the microwave plasma reaction, in which the weight percentages of C, H, O, N, and S were 38.65%, 5.58%, 0.20%, 0.00%, and 43.05%, respectively. The molar ratio of C:H:O in rice straw was 1:1.73:0.84, which is comparable to that of a cellulose monomer (chemical formula: $C_6H_{10}O_5$, where the molar ratio of C:H:O is 6:10.56:5.04), indicating that cellulose is the main ingredient of rice straw. After thermal decomposition as a result of the microwave plasma reaction, the weight percentages of C, H, O, N, and S were 49.61%, 4.56%, 0.36%, 0.00%, and 15.91%, respectively, and the molar ratio of C:H:O was estimated to be 1:1.10:0.24. Fig. 5 shows the SEM profiles of rice straw before and after the microwave plasma reaction. The morphology of rice straw changed to

Table 1
Heating rate and maximal temperature under different microwave power.

Power (W)	Heating rate (°C/s)	T_{max} (°C)
700	2.42	760
800	2.25	790
900	2.63	820
1000	2.75	849
1100	2.78	859

Table 2
Elemental analysis of rice-straw before and after microwave plasma reaction.

Element	Before		After	
	Weight ratio (%)	Molar ratio	Weight ratio (%)	Molar ratio
C	38.65	1.00	49.61	1.00
H	5.58	1.73	4.56	1.10
N	0.20	0.01	0.36	0.01
O	43.05	0.84	15.91	0.24



(a) (b)

Fig. 5. SEM profiles of rice-straw: (a) before and (b) after microwave plasma reaction.

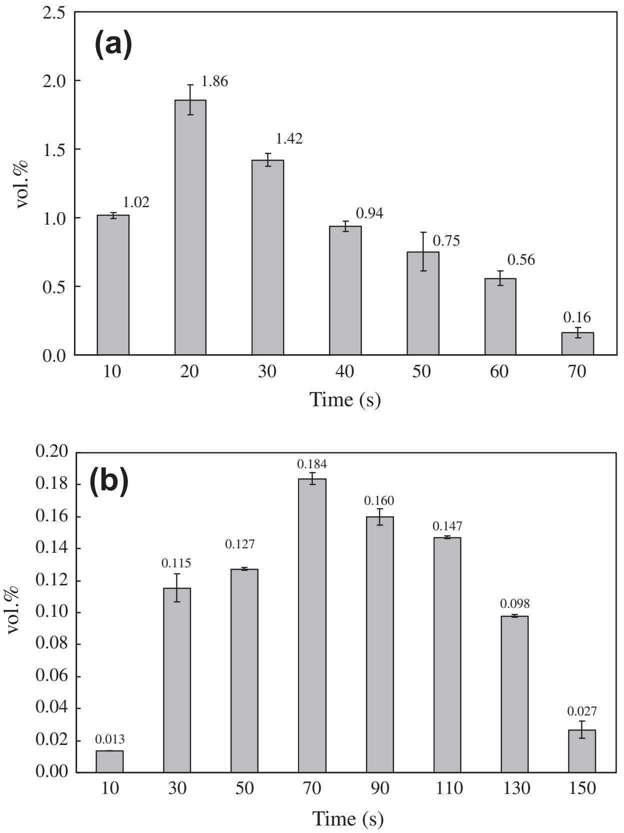


Fig. 6. The hydrogen concentration distribution of rice-straw at different periods: (a) upstream method and (b) downstream method. The microwave power is 800 W.

block and random shapes after the microwave plasma treatment, indicating that thermal decomposition is present after the microwave plasma reaction.

3.3. The hydrogen concentration analysis of rice-straw

3.3.1. Upstream method

Fig. 6a shows the hydrogen concentration distribution in rice straw at different periods when the upstream method was applied. The hydrogen concentrations were 1.02 ± 0.02%, 1.86 ± 0.11%, 1.42 ± 0.05%, 0.94 ± 0.04%, 0.75 ± 0.14%, 0.56 ± 0.05%, and 0.16 ± 0.04% at 10, 20, 30, 40, 50, 60, 70 s, respectively. When this method was used, the highest hydrogen concentration in rice straw occurred at 20 s; that is, the hydrogen concentration increased quickly from 0 to 20 s, then decreased after 20 s. Notably, the hydrogen concentration decreased quickly from 0 to 20 s, indicating that the decomposition was complete after 20 s. Fig. 7a shows the RGA absorption distribution of rice straw when the upstream method was used. The hydrogen production of rice straw after microwave plasma thermal decomposition was approximately 17.09%. After the microwave

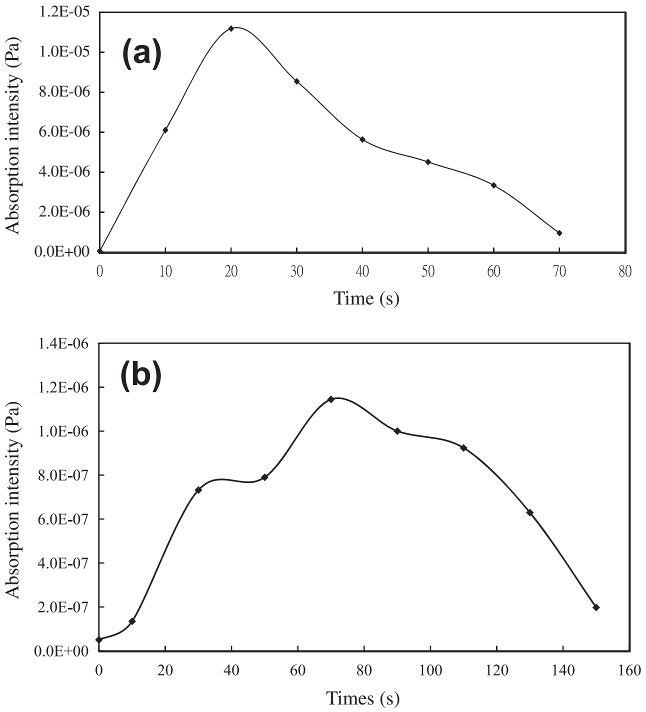


Fig. 7. The RGA absorption distribution of rice-straw: (a) upstream method and (b) downstream method. The microwave power is 800 W.

Table 3
The gas product analysis of rice-straw.

Ingredient (vol.%)	Upstream			Downstream		
	800 W	900 W	1000 W	800 W	900 W	1000 W
H_2	47.92	52.66	56.08	33.65	40.15	45.39
CO_2	24.40	19.41	16.43	29.99	24.57	23.23
CO	21.27	21.25	20.06	23.03	23.56	21.50
Other	6.41	6.68	7.43	13.34	11.71	9.88

plasma treatment, the results of GC/TCD measurement reveal that the major products of rice straw are H_2, N_2, CO_2, CO, and other trace gases. When the carrier gas N_2 is neglected, the concentrations of H_2, CO_2, and CO were 47.92%, 24.40%, and 21.27%, respectively, and the sum of H_2, CO_2, and CO is 93.59 vol.%. Table 3 shows that the average concentrations of H_2, CO_2, and CO at 800 W were 47.92 ± 2.87%, 24.40 ± 2.55% and 21.27% ± 3.04%, respectively, whereas that of other undetected gas at 800 W was only 6.41 ± 2.80%, indicating that most gases were detected and identified. Based on an analysis of GC-TCD results, the microwave plasma decomposition of rice straw produces H_2, and the concentration was 30 vol.%. Based on an analysis of GC/TCD results at 900 W and 1000 W, the detected gaseous products consisted primarily of H_2, N_2, CO_2, CO, and small amounts of other species. When nitrogen from the reaction product gas stream is neglected, the volume fractions of H_2, CO_2, and CO concentrations were 52.66%, 19.41%, and 21.25% at 900 W, respectively, and 56.08%, 16.43%, and 20.06%, respectively, at 1000 W; the sums of H_2, CO_2, and CO volume fractions were 93.31 vol.% and 92.56 vol.% at microwave power levels of 900–1000 W, respectively. Moreover, the concentrations of H_2, CO_2, and CO were 52.66 ± 3.06%, 19.41 ± 1.31%, and 21.25% ± 1.53% at 900 W, respectively, and 56.08 ± 1.57%, 16.43 ± 1.77%, and 20.06 ± 1.16% at 1000 W, respectively; those of the other undetected gas ingredients were 6.68 ± 1.75% and 7.43 ± 1.32% at 900 W and 1000 W, respectively. Based on an analysis of GC/TCD results, applying a high power of microwave plasma decomposes rice straw, and results in a higher hydrogen concentration than applying a low power does, when the upstream method is used.

3.3.2. Downstream method

Fig. 6b shows the hydrogen concentration distribution in rice straw when the downstream method was applied. The hydrogen concentrations were calculated based on the detection of absorption at 10, 30, 50, 70, 90, 110, 130, and 150 s, and were 0.013 ± 0.0001%, 0.115 ± 0.0087%, 0.127 ± 0.0009%, 0.184 ± 0.0037%, 0.160 ± 0.0049%, 0.147 ± 0.001%, 0.098 ± 0.0009%, and 0.027 ± 0.0055%, respectively. Fig. 7b shows the RGA absorption distribution of rice straw when the downstream method was used. The hydrogen production of rice straw resulting from microwave plasma thermal decomposition was approximately 6.68%. When this method was used, the hydrogen concentration of rice straw increased from 0 to 70 s, and decreased after 70 s. The highest hydrogen concentration thus occurred at 70 s, indicating that no hydrogen formed after 70 s. Hydrogen detected after 70 s was the remaining hydrogen in the pipe. Based on the results of GC/TCD analysis, the detected gaseous products consisted of H_2, N_2, CO_2, CO, and small amounts of other species. When the carrier gas of nitrogen is neglected, the concentrations of H_2, CO_2, and CO were 33.65%, 29.99%, and 23.03%, respectively, and the sum was 86.66 vol.%. As shown in Table 3, the gas product analysis of rice straw when the downstream method at 800 W was used shows that the average concentrations of H_2, CO_2, and CO were 33.65 ± 0.95%, 29.99 ± 2.38%, and 23.03 ± 2.69%, respectively, whereas that of other undetected gas was 13.34 ± 2.50%. Based on the analysis of GC/TCD results at 900 W and 1000 W, the detected gaseous products consisted primarily of H_2, N_2, CO_2, CO, and small amounts of other species. When nitrogen from the reaction product gas stream is neglected, the volume fractions of H_2, CO_2, and CO concentrations were 40.15%, 24.57%, and 23.56%, respectively, at 900 W, and 45.39%, 23.23%, and 21.50%, respectively, at 1000 W; the sum of H_2, CO_2, and CO volume fractions is 88.28 vol.% and 90.12 vol.% at microwave power levels of 900–1000 W, respectively. Moreover, the concentrations of H_2, CO_2, and CO were 40.15 ± 2.71%, 24.57 ± 1.17%, and 23.56 ± 1.17%, respectively, at 900 W and 45.39 ± 1.88%, 23.23 ± 3.32%, and 21.50 ± 0.56%, respectively, at 1000 W; however, the concentrations of the other undetected gas ingredients were 11.71 ± 2.40% and 9.88 ± 1.51% at 900 W and 1000 W, respectively. Based on the analysis of GC/TCD results, applying a high power of microwave plasma decomposes rice straw, and results in a higher hydrogen concentration than applying a low power does, when the downstream method is used.

3.4. Analysis of rice straw hydrogen production at various powers

Fig. 8a shows the effect of hydrogen concentration distribution in rice straw at various powers when the upstream method was used. The applied microwave powers, ordered from highest to lowest according to the resulting hydrogen concentration in rice straw, are presented as follows: 1000 W (2.78 ± 0.03%) > 900 W (2.52 ± 0.05%) > 800 W (1.85 ± 0.05%). The applied microwave powers, ordered from highest to lowest according the sum of hydrogenproduction, are presented as follows: 1000 W (27.66%) > 900 W (24.24%) > 800 W (17.09%). The hydrogen concentration in the rice straw products increased between 0 and 20 s, and the maximum occurred at 20 s when 800 W, 900 W, and 1000 W were applied. The hydrogen concentration decreased between 20 and 70 s at all power levels, and decreased rapidly at the 1000 W level between 20 and 30 s, indicating that the reaction of thermal decomposition decreases as the power increases. Fig. 8b shows the effects of hydrogen concentration distribution in rice straw at various powers when the downstream method was used. The applied microwave powers, ordered from highest to lowest according to the hydrogen concentration in rice straw, are presented in the following order: 800 W (0.184 ± 0.0037%) > 900 W (0.171 ± 0.0041%) > 1000 W (0.146 ± 0.005%). The hydrogen concentration of the rice straw products when the downstream method was used exhibited an irregular trend at various powers. This can be attributed to the rapid increase in the temperature of the cavity, the rapid increase in the pressure drop ($\triangle P$) between the inside and outside reactors, and the fast gas flow in the tube, disabling the RGA from detecting gases instantaneously.

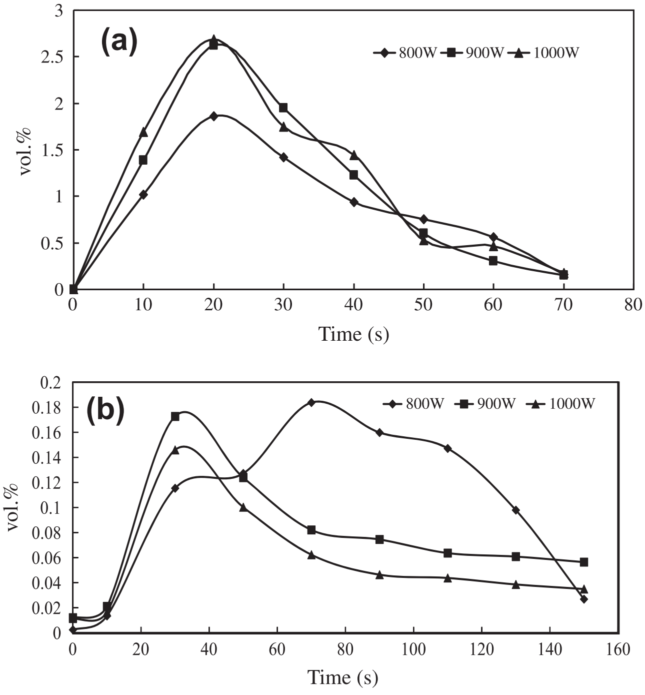


Fig. 8. The hydrogen concentration distribution of rice-straw at various powers (a) upstream method and (b) downstream method.

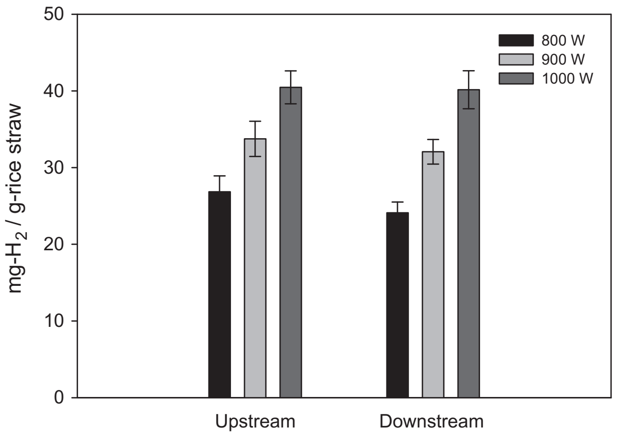


Fig. 9. Formation of H_2 in a unit mass of rice-straw.

Furthermore, we measure the total gaseous hydrogen mass yield per unit mass of rice straw consumed. The destroyed mass of biomass is the difference between the mass of rice straw before pyrolysis and the mass of pyrolyzed solid residues. The mass flow rate of production (ROP) of hydrogen is determined by:

$$\mathrm{ROP} = \left(\frac{12\ \mathrm{l}}{\mathrm{min}}\right) \times \left(\frac{1\ \mathrm{mole}}{24.5\ \mathrm{l}}\right) \times C_{\mathrm{gas}}(\%) \times \text{molecule weight} \qquad (3)$$

where 24.5 l is the ideal-gas volume for one mole at temperature of 298 K and C_{gas} is the mass fraction of H_2. The total volume flow rate of 12 slm that enters the reactor is the volume flow rate of nitrogen. The results, shown in Fig. 9, indicate that 1 g of rice straw can produce 26.85, 33.75, and 40.47 mg H_2 at 800 W, 900 W, and 1000 W, respectively, when the upstream method was used, whereas 1 g of rice straw can produce 24.11, 32.07, and 40.15 mg H_2 at 800 W, 900 W, and 1000 W, respectively, when the downstream method was used. The rice straw exhibited larger hydrogen production using upstream method than that of downstream method. This can be attributed to upstream feeding method is similar to continuous feeding, downstream feeding method is similar to batch feeding, therefore upstream feeding method is a key factor influencing the hydrogen production.

4. Conclusion

In this study, rice straw was pyrolyzed in a microwave oven in different situations. The detected gaseous products consisted of H_2, N_2, CO_2, CO, and small amounts of other species obtained from the conversion of biomass to hydrogen. The CO_2 concentrations of rice straw at 800 W, 900 W, and 1000 W when the upstream method was used were 24.39%, 19.41%, and 16.42%, respectively, and the CO_2 concentrations of rice straw at 800 W, 900 W, and 1000 W when the downstream method was used were 29.99%, 24.57%, and 23.23%, respectively. Regarding the feeding of rice straws into the microwave plasma system at 1000 W when the upstream method was used, the concentration of hydrogen was 56.08%, 40.47 mg/g of rice straw of hydrogen was produced, and the conversion rate was 67.45%; these results were determined to be optimal for hydrogen production.

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